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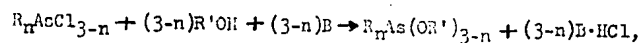
METHODS OF PREPARING ALKYL ESTERS AND GLYCOL ESTERS  
OF ARSENOUS AND ARYLARSENOUS ACIDS

Gil'm Kamay, Z. L. Khirnova,  
 and N. A. Chadayeva

Various methods for the preparation of alkyl and aryl esters of arsenous and arylarsenous acids have been described in chemical literature.

The simplest and most convenient methods for the synthesis of alkyl esters of arsenous and arylarsenous acids are those based on the action of arsenous acid anhydride or arylarsine oxide on alcohols or of chloroarsines on alcoholates.

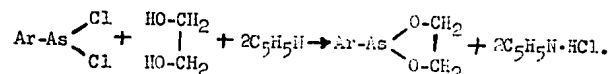
Another method which can be applied is one extensively used in our laboratory [7] based on the interaction of chloroarsines with alcohols in the presence of anhydrous pyridine or dimethylaniline according to the following scheme:



where B represents pyridine or dimethylaniline.

The present article describes comparative experimental data which were obtained by synthesizing glycolic esters of arsenous, phenylarsenous, and p-tolylarsenous acids according to two different methods.

First, we obtained glycol esters of arylarsenous acid through the action of arylidichloroarsine on glycols in the presence of pyridine according to the following scheme:



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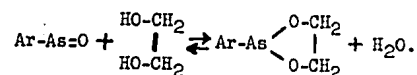
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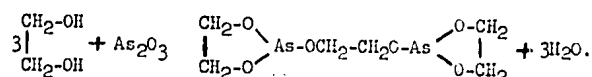
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Then, we obtained the same products by the interaction of glycols with arylarsine oxide by heating without dehydrating agents. This reaction proceeded according to the following scheme:



The physical properties and analytical data for the products obtained by the two methods are listed in the appended table. The table shows that the yield of products is considerably greater when the second method is used. In the last example, the yield reaches 85% and the isolated substances are chemically purer.

We also studied the preparation of the ethylene glycol ester of arsenous acid from arsenous acid anhydride and ethylene glycol according to the following equation:



The synthesis was carried out in the following manner: 60 g of arsenous acid anhydride and 86.4 g of ethylene glycol were placed in an Arbuzov distillation flask. The flask was heated on an oil bath at 150° for 15-20 minutes. Then, to remove water formed during the course of this reaction, an aspirator pump was connected with the apparatus. Further distillation of the products was done with the aid of an oil pump. After a twofold distillation, 80.2 g of a fraction boiling at 160-161° at 2 mm were obtained. The yield of pure ethylene glycol ester of arsenous acid was 92.4%.

We thus demonstrated that the simplest method for preparing glycolic esters of arsenous and arylarsenous acids is through the reaction of the corresponding glycol with arylarsine oxide or arsenous acid anhydride.

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No	Formula of Ester	Method	Boiling Point (°/mm)	d <sub>4</sub> <sup>20</sup> °	n <sub>D</sub> <sup>20</sup>	M <sub>P</sub> <sub>D</sub>		As (%)		Yield (%)
						Found	Calcu- lated	Found	Calcu- lated	
1	<chem>C6H5As(=O)(OC)OC</chem>	1	121-122/9	1.5365	1.6101	47.23	47.34	35.26	35.32	56.6
		2	122-122.5/9	1.5279	1.5961	47.22	47.34	35.06	35.32	64.5
2	<chem>C7H7As(=O)(OC)OC</chem>	1	138-138/10	m p	134-135°			32.74	33.13	45.6
		2	137-138/9	m p	134-135°			32.3	33.13	66.1
3	<chem>C6H5As(=O)(OC)OCCc1ccccc1</chem>	1	156-158/11	1.3584	1.5540	63.55	62.75	27.59	27.73	65.3
		2	158-159/11	1.3577	1.5512	63.49	62.75	27.59	27.73	74.3
4	<chem>C6H5As(=O)(OC)OCC</chem>	1	135-136/12	1.4618	1.5980	52.76	52.06	32.90	33.13	62.1
		2	133/10	1.4642	1.5990	52.75	52.06	32.53	33.13	69.0
5	<chem>C6H5As(=O)(OC)OCC1=CC=CC=C1</chem>	1	171-172/12	1.4591	1.5841	58.45	58.13	29.00	29.25	50.3
		2	155/2	1.4594	1.5895	58.43	58.13	29.42	29.25	59.8
6	<chem>C6H5As(=O)(OC)OC1=CC=CC=C1</chem>	1	155/2	m p	85-87°			28.13	28.79	20.1
		2	176-179/5	m p	85-86°			28.50	28.79	86.0

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